JC526 U.S. PTO
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09/22/98

UTILITY PATENT APPLICATION TRANSMITTAL

(Only for new nonprovisional applications under 37 CFR 1.53(b))

JC560 U.S. PTO
09/22/98

Attorney Docket No.	0163-0707-2X
First Named Inventor or Application Identifier	Kenji MIWA, et al.
Title	METHOD OF REFINEMENT OF MICROSTRUCTURE OF METALLIC MATERIALS

APPLICATION ELEMENTS

See MPEP chapter 600 concerning utility application contents

ADDRESS TO: **Assistant Commissioner for Patents**
Box Patent Applications
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1.■ Fee Transmittal Form
(Submit an original, and a duplicate for fee processing)

2.■ Specification [Total Pages 20]

3.■ Drawing [Total Sheets 1]

4. Oath or Declaration [Total Pages 4]
   - a.■ Newly executed (copy)
   - b.☐ Copy from a prior application (37 CFR 1.63(d))
     (for continuation/divisional with Box 17 completed)
     [Note Box 5 below]
     - i.☐ DELETION OF INVENTOR(S)
       Signed statement attached deleting inventor(s) named in the prior application,
       see 37 CFR 1.63(d)(2) and 1.33(b).

5.☐ Incorporation By Reference (*useable if Box 4b is checked*)
The entire disclosure of the prior application, from which a copy of the oath or declaration is supplied under Box 4b, is considered as being part of the disclosure of the accompanying application and is hereby incorporated by reference therein.

ACCOMPANYING APPLICATION PARTS

6. ☐ Assignment Papers (cover sheet & document(s))
7. ☐ 37 CFR 3.73(b) Statement ☐ Power of Attorney (*when there is an assignee*)
8. ☐ English Translation Document (*if applicable*)
9. ☐ Information Disclosure Statement (IDS)/PTO-1449 ☐ Copies of IDS Citations
10. ☐ Preliminary Amendment
11. ■ White Advance Serial No. Postcard
12. ☐ Small Entity Statement(s) ☐ Statement filed in prior app, Status still proper and desired
13. ☐ Certified Copy of Priority Document(s)
14. ■ Other: Notice of Priority

15. If a CONTINUING APPLICATION, *check appropriate box and supply the requisite information*:
☐ Continuation ☐ Divisional ☐ Continuation-in-part (CIP) of prior application No:

16. Amend the specification by inserting before the first line the sentence:
This application is a ☐ Continuation ☐ Division ☐ Continuation-in-part (CIP)
of application Serial No. , filed on .

17. CORRESPONDENCE ADDRESS

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Docket No: 0163-0707-2X

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor(s): Kenji MIWA, et al.

Serial No: New Application

Filing Date: HEREWITH

Title: METHOD OF REFINEMENT OF MICROSTRUCTURE OF METALLIC MATERIALS

FEE TRANSMITTAL

Assistant Commissioner For Patents
Washington, D.C. 20231

CLAIMS	(1) FOR	(2) NUMBER FILED	(3) NUMBER EXTRA	(4) RATE	(5) CALCULATIONS
	TOTAL CLAIMS	11 -20=	0	X $22=	$0.00
	INDEPENDENT	4 -3=	1	X $82=	$82.00
	MULTIPLE DEPENDENT CLAIMS (if applicable)			+ $270=	$270.00
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			BASIC FEE		$790.00
			TOTAL OF ABOVE CALCULATIONS =		$1,142.00
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Respectfully submitted,

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SPECIFICATION

METHOD OF REFINEMENT OF MICROSTRUCTURE OF METALLIC MATERIALS

Description of the Invention

This invention relates to a method for refining a microstructure of metallic materials. More particularly, the present invention relates to a method of refinement of microstructure of metallic materials characterized in that allows microstructure of metallic materials to be refined irrespective of the type of metal or refining agent, wherein high-energy vibration force such as electromagnetic vibrating force, ultrasonic vibrating force, or the like is applied directly to molten metallic materials. The present invention also relates to a method for refining solid metal particles by the above-described method to move them to a prescribed location.

Background of the Invention

Methods for refining microstructure of metallic materials are broadly classified into two types such that methods in which refining agents are added to

molten metallic materials to refine the microstructure of the metallic materials solidified, and methods in which the solid metallic materials are subjected to forming processes and heat treatments to refine the microstructure thereof.

Specifically, in the first group of the methods, refining agents act as nuclei for the solid metal crystal particles to be formed during solidification, yielding a refined microstructure that corresponds to the dispersion state of the refining agents, whereas in the second group of the methods, microstructures refined are obtained by recrystallization of the metals generated by heat treatments that follow forming processes such as rolling, extrusion, or the like.

In the methods of the first group, however, a close crystallographic relationship achieved between the refining agent and the solid crystal particles is required in order to allow the refining agent to be effective, and it is impossible to obtain adequate refining agents for some types of metals.

In addition, the refined structure smaller than the particle size of the refining agent cannot be made.

In the methods of the second group, it is difficult to yield adequate refining because forming processes such as rolling, extrusion and the like are limited in their effects, and exceeding these limits causes fracture of the metal, and there is a tendency to cause

metals recrystallized as well as metal particles enlarged as a result of the heat treatment that follows forming.

An urgent need therefore existed for developing a novel method for refining microstructure of metallic materials that would be able to solve the above-described problems of the conventional methods.

An objective of the present invention is to overcome these subjects.

Abstract of the Invention

The present invention provides a method for refining microstructure of metallic materials.

The present invention relates to a method which comprises forming cavitation (cavities) in molten metal by the application of high-energy vibrating force to a metal in the process of solidification, and crushing the newly formed solid crystal particles by the impact pressure generated during the collapse of the cavities to refine the microstructure of the material. High-energy electromagnetic vibrating force is applied to a solidifying metal sample 10 by the simultaneous imposition of an electric current and a magnetic field in an apparatus comprising an electromagnet 12 for applying a stationary magnetic field and an electrode 11 for passing an alternating current through the metal

sample, so that the solid crystal particles are crushed into small pieces, yielding a fine microstructure thereof.

Detailed Description of the Invention

Specifically, an objective of the present invention is to provide a novel method for refining microstructure of metallic materials that capable of refining the microstructure thereof irrespective of the type or composition of the metallic materials.

Another objective of the present invention is to provide a method for refining microstructure of metallic materials that facilitates refining even for metals that have been difficult to refine in the past.

Still another objective of the present invention is to provide a method for refining microstructure of metallic materials to move it to a prescribed location.

The following technological means are employed in the present invention, which is aimed at overcoming the aforementioned subjects.

(1) A method for refining microstructure of metallic materials, characterized in that comprises forming cavitation (cavities) in molten metal by the direct application of high-energy vibrating force such as electromagnetic vibrating force, ultrasonic vibrating force to the molten metal, crushing the resulting solid

metal crystal particles into small pieces by the impact pressure generated during the collapse of the cavities, and yielding a refined microstructure thereof.

(2) The method for refining microstructure of metallic materials according to (1) above, wherein the high-energy vibrating force is applied during the solidification of said metal.

(3) The method for refining microstructure of metallic materials according to (1) or (2) above, wherein the high-energy vibrating force is applied to a metal in the process of solidification by the simultaneous imposition of an electric current and a magnetic field to said molten metal or solidifying metal.

(4) A method for refining microstructure of metallic materials, characterized in that comprises forming cavitation (cavities) in molten metal by the direct application of high-energy vibrating force such as electromagnetic vibrating force, ultrasonic vibrating force to the molten metal, crushing solid particles of other metals, intermetallic compounds, or the like dispersed in the molten metal as well as the solid metal formed during solidification into small pieces by the impact pressure generated during the collapse of the cavities, and yielding refined microstructure thereof.

(5) A method for refining microstructure of metallic materials, characterized in that comprises forming

cavitation (cavities) in molten metal by the direct application of high-energy vibrating force such as electromagnetic vibrating force, ultrasonic vibrating force to the molten metal, crushing the solid particulate ceramics or other nonmetals dispersed in the molten metal as well as the solid metal formed during solidification into small pieces by the impact pressure generated during the collapse of the cavities, and yielding refined microstructure thereof.

(6) A method for refining solid metal particles formed during solidification to move them to a prescribed location by the simultaneous imposition of an electric current and a magnetic field on the molten metal in the process of final solidification.

(7) The method according to (6) above, wherein the solid metal particles formed during solidification are refined to shift them to a periphery of a tube by the simultaneous imposition of an electric current and a magnetic field on the molten metal in the process of final solidification.

(8) The method according to (6) above, wherein the solid particles of other metals, intermetallic compounds, or the like dispersed in molten metal as well as solid metal particles formed during solidification are refined to shift them to a periphery of a tube by the simultaneous imposition of an electric current and a magnetic field on the molten metal in the process of

final solidification.

(9) The method according to (6) above, wherein the solid particulate ceramics or other nonmetals dispersed in molten metal as well as solid metal particles formed during solidification are refined to shift them to a periphery of a tube by the simultaneous imposition of an electric current and a magnetic field on the molten metal in the process of final solidification.

(10) The method according to (6) above, wherein the solid particles dispersed in molten metal are refined to move them to a location separated from the location of the initial dispersed state by the simultaneous imposition of an electric current and a magnetic field.

The present invention will now be described in detail.

The invention of this application is characterized in that the microstructure of metallic materials is refined by the direct application of high-energy vibrating force to them. In this case, it is important that electric current and magnetic field be simultaneously applied as the high-energy vibrating force, whereas applying the electric current or magnetic field alone has no significant effect on the fine microstructure of metallic materials. The reason is that the electromagnetic vibrating force is a Lorentz force that can only be generated when an electric

current and a magnetic field are applied simultaneously.

Electromagnetic vibrating force and ultrasonic vibrating force are exemplified as specific examples of high-energy vibrating force, but these examples are not all-encompassing and include all other types of force capable of exerting high-energy vibrating force on molten metal in the same manner.

The high-energy vibrating force is applied to molten metal, in which case it is preferable for the high-energy vibrating force to be applied to solidifying metal.

As used herein, the term "molten metal" refers to a metal that is completely liquefied which kept at a temperature above its melting point. In addition, the term "solidifying metal" refers to a liquid metal containing solid metal crystals that form at a temperature below the melting point.

The present invention can be adequately applied, for example, to aluminum alloys such as Al-Si alloys or magnesium alloys, but a distinctive feature of the present invention is that it allows any refining agent or metal to be used, and that, in particular, there is no dependence on the type or composition of metal.

When high-energy vibrating force is applied to a solidifying metal in accordance with the above-described method, the microstructure thereof is refined by

forming cavitation (cavities) in the molten metal and allowing the impact pressure generated during the collapse of the cavities to crush the resulting solid metal crystal particles into small pieces.

Because cavitation is induced while some of the metal is still in the molten state, not only the newly formed solid metal crystals but also the already existing solid metal particles are crushed by the application of high-energy vibrating force until the molten metal has completely solidified, making it possible to obtain a refined microstructure thereof.

A solidified microstructure of metallic materials can therefore be refined as well.

The high-energy vibrating force should be applied during (in the process of) solidification. It is difficult to form cavitation (cavities) when high-energy vibrating force is applied to metallic materials after solidification thereof, and therefore there is a possibility that the solid metal crystal particles will not be crushed.

In addition, in this invention, even metals that are difficult to refine by conventional methods can be readily refined because the refining effect of this invention by the high-energy vibrating force does not depend on the type or composition of the metal.

Silicon crystals as initially crystallized particles in a hypereutectic aluminum-silicon alloy,

can, for example, be refined to a crystal particle diameter of 0.5-3.0 μm by the method for refining microstructure of metallic materials through application of high-energy vibrating force in accordance with the present invention.

The present invention also allows solid particles of other metals, intermetallic compounds, or the like, as well as solid particulate ceramics or other nonmetals dispersed in molten metal to be crushed in the same manner as the solid metal formed during solidification.

The method of the present invention allows, for example, 20- to 30-μm silicon carbide particles dispersed in an aluminum alloy to be refined to a size of 0.1-2.0 μm.

Another feature of the present invention is that the solid metal particles formed during solidification can be refined to move them to a prescribed location by the simultaneous application of electric current and magnetic field to the molten metal in the process of final solidification thereof. Specifically, the solid metal formed during solidification can be refined to shift it to the periphery of a cylindrical tube or container disposed such that the axial direction of the cylinder is orthogonal to the magnetic field; solid particles of other metals, intermetallic compounds, or the like, as well as solid particulate ceramics or other nonmetals dispersed in molten metal can be shifted

in the same manner as the solid metal formed during solidification to the periphery of a cylindrical tube or container disposed in the same manner as the solid metal; and the aforementioned solid particles can be refined to move them to a separate location the inside tube or container from the location of the initial dispersed state. Another specific feature is that the shifting locations can be concentrated in the end portion of a sample by moving the sample within the magnetic field.

Brief Description of the Drawings

Fig. 1 is a schematic view illustrating an example of an apparatus suitable for implementing the present invention.

Description of marks

10	metal sample
11	electrode
12	electromagnetic coil

Examples

The present invention will now be described in detail through examples thereof, but the present invention is not limited by these examples.

Fig. 1 shows an example of the apparatus for

implementing the present invention. In the drawing, 10 is a metal sample, 11 is an electrode disposed in contact therewith, and 12 is an electromagnetic coil disposed such that it envelops the metal sample.

When an alternating current of about 80 A is passed through the metal sample via the electrode, the metal sample is melted by Joule heat generated, and the temperature of the metal sample reaches a prescribed temperature. The temperature of the molten metal sample is then lowered and solidification of the metal sample is started by reducing the electric current. An electromagnetic vibrating force based on the alternating current and a stationary magnetic field is created by the application of a stationary magnetic field of 1.4 T (Tesla) through the intermediary of the electromagnet 12, and at this time the molten metal sample is vibrated by the vibrations. As a result, cavities are formed in the metal sample, and the solidified metal crystals are crushed by the cavitation phenomenon.

The above-described apparatus was used to impose electromagnetic vibrating force upon a solidifying alloy in the form of a hypereutectic Al-17% Si alloy. The results are shown in Table 1. As shown in Table 1, it was found that the silicon particles initially crystallized were crushed into small pieces.

Table 1

		Crystal grain diameter (μ m)
Example of present invention	Introduction of high vibrational energy	0.5 - 3
Conventional example	Use of refining agents	30 - 50

(Examples of the inventions defined in Claims 4-5)

The above-described apparatus was used to apply electromagnetic vibrating force to a solidifying aluminum alloy and to solidifying zinc in order to refine silicon carbide particles dispersed in the aluminum alloy and to refine Fe_3P compound particles dispersed in the zinc. The results are shown in Table 2. It was found that the dispersed silicon carbide particles and Fe_3P compound particles were crushed into small pieces.

Table 2

	Diameter of Fe_3P particles in zinc (μ m)	Diameter of SiC particles in aluminum alloy (μ m)
Example of present invention	10 - 1	2 - 0.1
Conventional dispersant	50 - 100	20 - 30

(Examples of the inventions defined in Claims 6-10)

Electromagnetic vibrating force was applied to an Al-17% Si alloy in the process of final solidification in order to refine the alloy. As a result, the refined silicon particles as initially crystallized in a uniformly dispersed sample could be moved to the surface of the surrounding walls of a cylindrical tube. In addition, an alloy obtained by dispersing Fe_3P particles in zinc, and an alloy obtained by dispersing SiC particles in an aluminum alloy could also be moved to the surface of the surrounding walls of the cylindrical tube in the same manner as in the case of the Al-17% Si alloy.

Examples of the present invention have been described in detail above, but these examples merely serve as an illustration, and the same effect can be achieved for other metals, alloys, intermetallic compounds, semimetals, nonmetals, and the like. The present invention allows embodiments incorporating various changes based on the knowledge possessed by those skilled in the art to be implemented as long as these changes remain within the scope of the present invention.

The present invention relates to a method for refining microstructure of metallic materials characterized in that comprises forming cavitation (cavities) in molten metal by the direct application of high-energy vibrating force such as electromagnetic

vibrating force, ultrasonic vibrating force to the molten metal, and crushing the resulting solid metal crystal particles into small pieces by the impact pressure generated during the collapse of the cavities, and yielding a refined microstructure of the metal. The present invention allows microstructure of metallic materials to be readily refined to the level of fine particles without the use of refining agents and without any relation to the type or composition of the metal. It is also possible to refine solid particles of other metals, intermetallic compounds, or the like dispersed in the molten metal. It is further possible to shift solid metal particles and solid particles dispersed in molten metal toward the periphery of a tube or container.

Claims

1. A method for refining microstructure of metallic materials, characterized in that comprises forming cavitation (cavities) in molten metal by the direct application of high-energy vibrating force such as electromagnetic vibrating force, ultrasonic vibrating force to the molten metal, crushing the resulting solid metal crystal particles into small pieces by the impact pressure generated during the collapse of the cavities, and yielding a refined microstructure thereof.

2. The method for refining microstructure of metallic materials according to Claim 1, wherein the high-energy vibrating force is applied during the solidification of said metal.

3. The method for refining microstructure of metallic materials according to Claim 1 or 2, wherein the high-energy vibrating force is applied to a metal in the process of solidification by the simultaneous imposition of an electric current and a magnetic field to said molten metal or solidifying metal.

4. A method for refining microstructure of metallic materials, characterized in that comprises forming cavitation (cavities) in molten metal by the direct

application of high-energy vibrating force such as electromagnetic vibrating force, ultrasonic vibrating force to the molten metal, crushing solid particles of other metals, intermetallic compounds, or the like dispersed in the molten metal as well as the solid metal formed during solidification into small pieces by the impact pressure generated during the collapse of the cavities, and yielding refined microstructure thereof.

5. A method for refining microstructure of metallic materials, characterized in that comprises forming cavitation (cavities) in molten metal by the direct application of high-energy vibrating force such as electromagnetic vibrating force, ultrasonic vibrating force to the molten metal, crushing the solid particulate ceramics or other nonmetals dispersed in the molten metal as well as the solid metal formed during solidification into small pieces by the impact pressure generated during the collapse of the cavities, and yielding refined microstructure thereof.

6. A method for refining solid metal particles formed during solidification to move them to a prescribed location by the simultaneous imposition of an electric current and a magnetic field on the molten metal in the process of final solidification.

7. The method according to Claim 6, wherein the solid metal particles formed during solidification are refined to shift them to a periphery of a tube by the simultaneous imposition of an electric current and a magnetic field on the molten metal in the process of final solidification.

8. The method according to Claim 6, wherein the solid particles of other metals, intermetallic compounds, or the like dispersed in molten metal as well as solid metal particles formed during solidification are refined to shift them to a periphery of a tube by the simultaneous imposition of an electric current and a magnetic field on the molten metal in the process of final solidification.

9. The method according to Claim 6, wherein the solid particulate ceramics or other nonmetals dispersed in molten metal as well as solid metal particles formed during solidification are refined to shift them to a periphery of a tube by the simultaneous imposition of an electric current and a magnetic field on the molten metal in the process of final solidification.

10. The method according to Claim 6, wherein the solid particles dispersed in molten metal are refined to move them to a location separated from the location of

the initial dispersed state by the simultaneous imposition of an electric current and a magnetic field.

Abstract

The present invention provides a method for refining microstructure of metallic materials and the present invention relates to a method in which cavitation (cavities) is formed in molten metal by the application of high-energy vibrating force to a metal in the process of solidification, and the newly formed solid crystal particles are crushed by the impact pressure generated during the collapse of the cavities to refine the microstructure of the material, and high-energy electromagnetic vibrating force is applied to a solidifying metal sample 10 by the simultaneous imposition of an electric current and a magnetic field in an apparatus comprising an electromagnet 12 for applying a stationary magnetic field and an electrode 11 for passing an alternating current through the metal sample, so that the solid crystal particles are crushed into small pieces, yielding a fine microstructure thereof.

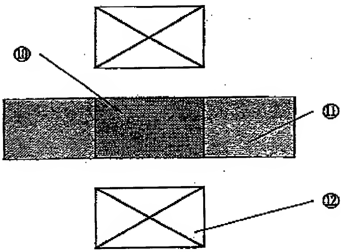

Fig. 1

Declaration and Power of Attorney For Patent Application

特許出願宣言書及び委任状

Japanese Language Declaration

日本語宣言書

下記の氏名の発明者として、私は以下の通り宣言します。

As a below named inventor, I hereby declare that:

私の住所、私書箱、国籍は下記の私の氏名の後に記載された通りです。

My residence, post office address and citizenship are as stated next to my name.

下記の名称の発明に関して請求範囲に記載され、特許出願している発明内容について、私が最初かつ唯一の発明者（下記の氏名が一つの場合）もしくは最初かつ共同発明者（下記の名称が複数の場合）であると信じています。

金属組織微細化法

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled.

METHOD OF REFINEMENT OF MICROSTRUCTURE OF METALLIC MATERIALS

上記発明の明細書は、

☒ 本書に添付されています。

☐ ___月___日に提出され、米国出願番号または特許協定条約国際出願番号を__________とし、
(該当する場合) __________に訂正されました。

the specification of which

☒ is attached hereto.

☐ was filed on __________ as United States Application Number or PCT International Application Number __________ and was amended on __________ (if applicable).

私は、特許請求範囲を含む上記訂正後の明細書を検討し、内容を理解していることをここに表明します。

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

私は、連邦規則法典第37編第1条56項に定義されるとおり、特許資格の有無について重要な情報を開示する義務があることを認めます。

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56.

Japanese Language Declaration
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私は、米国法典第35編119条（a）－（d）項又は365条（b）項に基づき下記の、米国以外の国の少なくとも一ヵ国を指定している特許協力条約365（a）項に基づく国際出願、又は外国での特許出願もしくは発明者証の出願についての外国優先権をここに主張するとともに、優先権を主張している、本出願の前に出願された特許または発明者証の外国出願を以下に、枠内をマークすることで、示しています。

I hereby claim foreign priority under Title 35, United States Code, Section 119 (a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or Section 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application(s)
外国での先行出願

(Number) (番号)	(Country) (国名)	(Day/Month/Year Filed) (出願年月日)	Priority Claimed 優先権主張	
9-275330	JAPAN	22/09/1997	☒ Yes はい	☐ No いいえ
			☐ Yes はい	☐ No いいえ

私は、第35編米国法典119条（e）項に基づいて下記の米国特許出願規定に記載された権利をここに主張いたします。

I hereby claim the benefit under Title 35, United States Code, Section 119(e) of any United States provisional application(s) listed below.

(Application No.) (出願番号)	(Filing Date) (出願日)	(Application No.) (出願番号)	(Filing Date) (出願日)

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I hereby claim the benefit under Title 35, United States Code, Section 120 of any United States application(s), or Section 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of Title 35, United States Code Section 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of application.

(Application No.) (出願番号)	(Filing Date) (出願日)	(Status: Patented, Pending, Abandoned) (現況：特許許可済、係属中、放棄済)

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Japanese Language Declaration
(日本語宣言書)

委任状：私は下記の発明者として、本出願に関する一切の手続きを米特許商標局に対して遂行する弁理士または代理人として、下記の者を指名いたします。
(弁護士、または代理人の指名及び登録番号を明記のこと)

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith: *(list name and registration number)*

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（第六またはそれ以降の共同発明者に対しても同様な情報および署名を提供すること。）

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